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Improved sample preparation method for SHRIMP analysis of delicate mineral grains exposed in thin sections

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Abstract: A new method is presented for preparing samples for contextual ion-microprobe isotopic studies of polished thin sections. Selected areas of one or more sections are cored using 4 to 8 mm diameter, diamond-tipped drill bits. A 5 mm diameter epoxy cylinder is prepared that exposes polished pieces of the analytical standard at one end. The thin-section disks and prepolished standard plug are together cast in epoxy. No further sample preparation is required, thereby preserving the original surface of the thin-section disks. This method improves the efficiency of carrying out in situ SHRIMP analysis by overcoming the typical limitation of finding few suitable minerals exposed on any given thin section, and obviates technical difficulties associated with using this medium.

Résumé : Dans le cadre d'études isotopiques à la microsonde ionique, on présente une nouvelle méthode de préparation des échantillons destinés à des mesures en milieu réel sur des lames minces polies. À l'aide d'un foret à couronne garnie de diamants d'un diamètre intérieur de 4 à 8 mm, on procède au carottage de zones choisies d'une ou de plusieurs lames minces. Parallèlement, on prépare un cylindre d'époxy de 5 mm de diamètre qui présente, à une extrémité, l'étalon d'analyse à surface préalablement polie. Les disques de lames minces et l'étalon poli sont enchâssés par la suite dans l'époxy. Les échantillons ne requièrent plus aucune autre forme de préparation. La surface originale des disques de lames minces est ainsi préservée. Cette méthode permet d'améliorer l'efficacité de l'analyse par des mesures localisées à la microsonde ionique SHRIMP en surmontant la difficulté caractéristique que pose la rareté de surfaces exposées de minéraux appropriés sur une lame mince donnée. Elle permet aussi d'éviter les difficultés techniques associées à l'utilisation de ce support.

INTRODUCTION

One of the strengths of the Sensitive High Resolution Ion Microprobe (SHRIMP) is its ability to carry out in situ (i.e. contextual) isotopic studies (e.g. Stern and Berman, 2000). The SHRIMP II at the Geological Survey of Canada is capable of accepting standard polished thin sections (2.7 cm by 4.5 cm), but their extensive use has been limited by a number of problems, including the need for specialized preparation, technical issues related to analysis and handling of this medium in the ion microprobe, and inefficiencies related to the limited number of suitable targets commonly exposed. The method presented here overcomes many of these difficulties.

PROBLEMS WITH THIN SECTIONS

Discrimination of secondary U and Pb ions in the ion microprobe depends on a number of factors, one of which is a subtle effect exerted by the surface of each individual mount. Consequently, calibration of a mineral standard is required to determine U-Pb ages for each prepared sample, be it a grain mount or thin section. Fragments of the standard must be embedded in the thin section, a rather difficult procedure that involves drilling a small depression, fixing the standard grains using epoxy, and spot-polishing back to the level of the thin section. Great care must be taken not to disturb other areas of the thin section that expose the minerals of interest, which are often quite small. It is very difficult to polish the standard without developing relief.

The location of the grains of interest within the section is critical, as the thin section is clamped within a specially designed steel frame during analysis. Only those grains within a central circular area, measuring approximately 1.5 cm in diameter and well removed from the edges of the steel frame, are actually accessible, as analyses near the edges will be adversely affected. Due to the restrictions in the accessible area of the thin section, and the fact that a thin section rarely exposes many suitable grains, the number of targets available for analysis is often less than 10. In order to collect a meaningful data set, more than one section may be needed. Each section requires laborious preparation and a separate calibration of the standard, a process that consumes 2 to 6 hours of analytical time. Use of thin sections tends to be very inefficient in terms of the number of grains and analyses acquired in a given session, and is thus an expensive proposition.

Vertical transfer of thin sections between the SHRIMP sample lock and source chamber is done by robotic arm manipulation. Thin sections, or more precisely their steel holders, present special problems during this procedure, particularly the maintenance of proper alignment. The thin sections may rotate about a horizontal axis, occasionally making it impossible to position them securely in the source chamber. This situation requires venting of the sample lock, realignment of the thin section, and a subsequent attempt at sample transfer. Although improvements to the thin section holder and transfer process are possible, the issues of inefficiency described above would still remain.

TECHNIQUE

In brief, with the new method, areas of interest from one or more thin sections are cored and cast, along with a polished plug containing pieces of calibration standard, in a 2.5 cm diameter epoxy mount.

Conventional thin sections are examined petrographically and by scanning electron microscopy to identify the targets and their textural setting; their locations are then marked on the polished side with waterproof ink. Marking the targets is most easily done using a special Nikon® objective marker, which temporarily replaces one of the objective lenses on a petrographic microscope. The target is centred under the crosshairs of the microscope, then the marker is moved into position. By pressing down on the outer ring of the marker, a circle (2 mm diameter) is imprinted on the thin section. A small (8 in.) standard drill press and four diamond-tipped coring drill bits with inner diameters of 4, 5, 6, and 8 mm (purchased from a local glass cutting company) are used to drill out the areas of interest. Experiments with a 2 mm bit were not successful, as the resulting small disks were very difficult to handle and the damage zone around their edges was large with respect to overall disk size. Consequently, the minimum recommended core diameter is 4 mm. Larger bits are used in cases where multiple targets are close to each other.

Using electrical tape, the thin section is affixed securely, rock side up, to a block of white Teflon™ (wood also works fine), in turn held in place by a customized aluminum clamp that serves to add sufficient mass to the assembly for subsequent subaqueous drilling. In early experiments, the thin section was held in place by a clamp that pressed on its edges, but this arrangement commonly resulted in breakage of the thin section. The thin section and holder are submerged in a clear acrylic box, in this case a Sigma-Aldrich™ storage box for β-emitters, containing cool tap water. The target is aligned under the bit and, using slow constant pressure, the bit is drilled (3100 rpm) into the thin section until a change in resistance indicates that the thin section has been drilled through. The core is removed by tapping gently on the bit, or by pushing the disk out through the hollow bit, in order not to scratch the rock surface. It is possible to drill 5 to 10 disks from a single thin section, depending on the size of bit used. Occasionally there is breakage of the thin section, but such fragments can also be easily drilled. Delamination of the rock section from the glass has also been observed in some cases.

The standard minerals are prepared using the conventional approach for 2.5 cm diameter epoxy mounts. Several clusters of three or four grains of the standard are placed on double-sided tape, cast in Struers Epofix™ epoxy, and polished following routine procedures (Stern, 1997). Subsequently, the polished mount is clamped into the aluminum holder (polished side up) and submerged in water, and individual clusters are drilled out using the 5 mm bit. Again, the

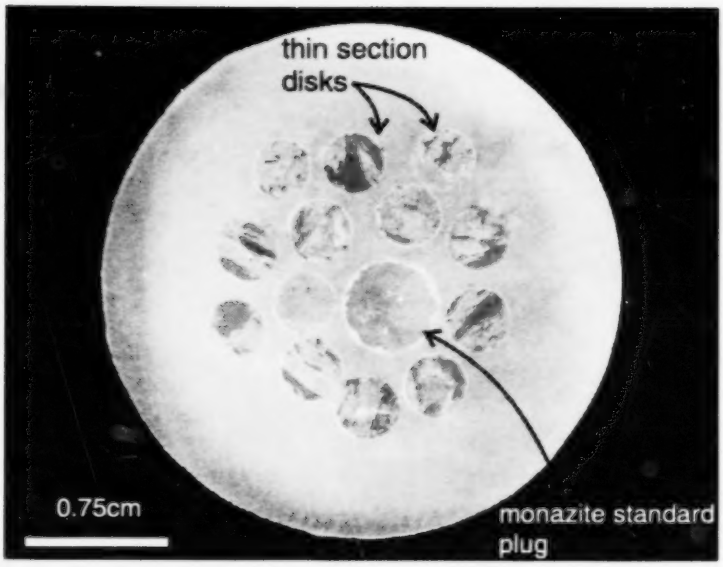

Figure 1.

Photograph of SHRIMP mount prepared for in situ monazite analysis using the technique outlined in the text. The plug containing the monazite standard is highlighted with an arrow.

epoxy plug is pushed out through the drill bit so as not to damage the polished surface. The result is a 5 mm diameter cylinder of epoxy with polished pieces of the standard exposed on one end.

The thin-section disks and standard plug are arranged, polished surfaces down, on double-sided tape. The disks and plug are positioned such that all the grains of interest fall within a 1.5 cm diameter circle in the centre of the mount, an area termed the mount 'bullseye'. Up to thirteen 4 mm disks, plus the standard plug, can be accommodated. Epoxy is poured over and around the disks and standard, and left to cure overnight. After the epoxy has set, the mount is cleaned with ethanol to remove any tape residue, then with soap and water. No further preparation or polishing is necessary (or desirable, in order to preserve the surface).

Prior to analysis, the mount is photographed in reflected light, as these images are ideal for locating grains during the analytical session. Following a second cleaning with soap and water, the mount is coated evaporatively with approximately 15 nm of high-purity gold. The mount can then be imaged with a scanning electron microscope.

CONCLUSIONS

Figure 1 is an example of a mount prepared using this new technique. The mount is composed of the 13 best minerals from 11 different thin sections, and illustrates the increased efficiencies achievable for in situ analysis. The resources required to collect the same amount of data from each individual thin section would have been prohibitive. Experience with isotopic analysis of these mounts indicates that there is no distinction from conventional grain mounts in terms of instrument performance and data quality. An individual thin section is now used only if numerous grains are exposed in its central area. For such cases, the preparation method has been improved as follows: a suitable hole is drilled through the section, which is then placed face down on the double-sided tape, and a prepolished standard plug is epoxy-cast into the hole.

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